

TREATMENT OF NATURAL RUBBER WASTEWATER USING OIL PALM
EMPTY FRUIT BUNCH, KAOLIN AND ZEOLITE AS COMPOSITE
ADSORBENT

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A thesis submitted in
fulfilment of the requirement for the award of the
Doctor of Philosophy

Faculty of Civil and Environmental Engineering
Universiti Tun Hussein Onn Malaysia

APRIL, 2018

DEDICATION

*To my beloved mother, Nusibah Mahat
And father Allahyarham Mangsor Bin Mat Aris
For her patient, encouragement and full support*

*To my beloved siblings,
Nazlina, Nazlita, Nazlisyam, Nazlizul and Ainul Anisah,
For constant support, understanding & love.*

*To my Lecturers and Friend
For giving me infinite care and blessing.
Thank you for your endless support for me.*



PTTAUTHM
PERPUSTAKAAN TUNKU TUN AMINAH

ACKNOWLEDGEMENT

In the name of Allah, the Most Beneficent and the Most Merciful. All praises to Allah the Almighty for giving me the strengths, guidance and patience in completing this thesis. First and foremost, I would like to express my deepest gratitude to my family for their endless love and support for me to pursue my doctorate degree. My deepest appreciation goes to Associate Professor Dr Zawawi Bin Daud for serving as a supervisor, Associate Professor Dr Aeslina Abd Kadir and Associate Professor Dr Azhar Abd Halim as co-supervisor in this research project and for providing me with the insights and guidance to recognize my mistake. Their understanding, wide knowledge and personal guidance have provided a good basis for the present thesis.

I would like to express my grateful thanks to Mr Bahtiar, Mr Kassim, Mrs Nadia, Mrs Fazlyana, Mrs Hazliana, Mr Azwan, and Mrs Mahfuzah for their technical guidance and support towards my experimental work in the laboratories at Faculty of Civil and Environmental Engineering University Tun Hussein Onn. Besides, special acknowledgement goes to research colleague; Mr Baharin Ahmad, Mr Khairudin Sakury, Dr Arif Rosli, Dr Paran Gani, Ms Norain Suhani, and a team of Centre of Advanced Research for Integrated Solid Waste Management (CARISMA) for sharing their knowledge, professional advice and guidelines throughout the whole project. Last but not least, I would like to express the deepest gratitude to Ministry of Higher Education for the scholarship received under MyBrain 15. Also, those who have directly and indirectly contributed to the accomplishment of this project, especially my friends for their motivation, encouragement and moral support. Thank you so much.

ABSTRACT

The development of composite adsorbents is a new technique applied in wastewater treatment. A combination of few adsorbents is proven to be capable of removing pollutants simultaneously compared to a single adsorbent. Thus, the aim of this study is to produce a composite adsorbent from natural zeolite, oil palm empty fruit bunch (EFB) and kaolin bound to chitosan to adsorb colour, chemical oxygen demand (COD) and ammoniacal nitrogen ($\text{NH}_3\text{-N}$) from natural rubber wastewater. Batch and fixed-bed adsorption techniques were used in this study. The combination ratio of zeolite, EFB and kaolin as a composite was optimised using D-optimal mixture design (DMD) while the characterization of composite adsorbents was done using Fourier Transform Infrared (FTIR), Field Emission Scanning Electron Microscopy (FESEM), Brunauer-Emmett-Teller (BET) and zeta potential. Batch experiments were carried out to determine the influence of various factors namely adsorbent dosage, pH, shaking speed and contact time of adsorption colour, COD and $\text{NH}_3\text{-N}$ on the composite adsorbent. Langmuir and Freundlich models were used to investigate isotherm adsorption while pseudo-first order, pseudo-second order, intra-particle diffusion and Elovich were used to examine kinetic behaviour. The fixed-bed adsorption performances were evaluated by varying the influent flow rate (2, 2.5 and 3 mL/min) while the adsorption kinetics was analysed using Thomas, Yoon-Nelson and Adam-Bohart kinetic models. The regeneration of the composite adsorbent for up to five adsorption/desorption cycles was also investigated. According to the results, the composition of 0.4g zeolite, 0.8g EFB and 0.8g kaolin was the best ratio in terms of colour, COD and $\text{NH}_3\text{-N}$ removal from natural rubber wastewater. FTIR and FESEM analyses before and after adsorption revealed that ion exchange was the main mechanism involved. The BET surface area of the composite adsorbent was $55.30 \text{ m}^2/\text{g}$. Meanwhile, negative values of zeta potential showed promising results for the adsorption process. The best conditions for the effective adsorption of colour, COD and $\text{NH}_3\text{-N}$ from natural rubber wastewater onto composite adsorbents were found to be 3g of adsorbent pH 7, 150

rpm shaking speed and 100 min contact time. The maximum removal of colour, COD and $\text{NH}_3\text{-N}$ using composite adsorbents from natural rubber wastewater was 98%, 87.2% and 94.2% respectively. The investigation of adsorption isotherms model showed that the adsorption isotherm data fitted well to the Langmuir isotherm. This indicated that the monolayer coverage on the composite adsorbent was dominant. On the other hand, the adsorption kinetics complied well with the pseudo-second-order kinetic model which indicated that the rate of the sorption reaction was controlled by the second-order mechanism (chemisorption). Column adsorption demonstrated that the removal of colour, COD and $\text{NH}_3\text{-N}$ can reach up to 99.99% at a lower flow rate of 2 ml/min which results in a longer breakthrough and exhaustion time. Meanwhile, the experiment data was found to comply well with the Thomas and Yoon-Nelson model rather compared to the Adam-Bohart model. The regeneration study achieved three (3) adsorption cycles of the spent composite adsorbent. In conclusion, the present study was able to prove that composite adsorbent is a promising adsorbent for the removal of colour, COD and $\text{NH}_3\text{-N}$ from natural rubber wastewater.



ABSTRAK

Pembangunan bahan penjerap komposit merupakan kaedah baru dalam aplikasi rawatan air sisa. Gabungan beberapa bahan penjerap terbukti mampu menyingkirkan bahan pencemar serentak berbanding dengan satu bahan penjerap sahaja. Tujuan kajian ini adalah untuk menghasilkan bahan penjerap komposit dari zeolit semulajadi, tandan kosong kelapa sawit (EFB) serta kaolin dan diikat menggunakan kitosan untuk menjerap warna, permintaan oksigen kimia (COD) dan ammonia nitrogen ($\text{NH}_3\text{-N}$) dari air sisa getah asli. Kaedah penjerapan kelompok dan turus lapisan tetap digunakan dalam kajian ini. Nisbah gabungan zeolit, EFB dan kaolin sebagai komposit ditentukan menggunakan kaedah reka bentuk campuran D-optimal (DMD), manakala pencirian bahan penjerap komposit dibuat menggunakan spektroskopi Inframerah (FTIR), mikroskop elektron pengimbas pancaran medan (FESEM), Brunauer-Emmett-Teller (BET) dan keupayaan zeta. Ujian kelompok dijalankan untuk menentukan pengaruh pelbagai faktor iaitu dos bahan penjerap, pH, kelajuan goncangan dan sentuhan masa bagi penjerapan warna, COD dan $\text{NH}_3\text{-N}$ oleh bahan penjerap komposit. Model Langmuir dan Freundlich digunakan untuk menyiasat isoterma penjerapan, manakala pseudo-tertib pertama, pseudo-tertib kedua, pembauran intra-partikel dan Elovich untuk meneliti sifat kinetik. Prestasi penjerapan lapisan tetap dinilai dengan mempelbagaikan kadar aliran influen (2, 2.5 dan 3 mL/min), manakala kinetik penjerapan dianalisa menggunakan model kinetik Thomas, Yoon-Nelson dan Adam-Bohart. Penjanaan semula bahan penjerap komposit untuk sehingga lima pusingan penjerapan/penyaherapan juga disiasat. Berdasarkan keputusan, komposisi zeolit 0.4g, kaolin 0.8g EFB and 0.8g ialah nisbah terbaik dalam penyingkiran warna, COD dan $\text{NH}_3\text{-N}$ dari air sisa getah asli. Analisis FTIR dan FE-SEM sebelum dan selepas penjerapan mendedahkan bahawa penukaran ion ialah mekanisma utama yang terlibat. Luas permukaan BET bahan penjerap komposit ialah $55.30 \text{ m}^2/\text{g}$. Sementara itu, nilai negatif keupayaan zeta menunjukkan potensi dalam proses penjerapan. Keadaan terbaik untuk penjerapan warna, COD, $\text{NH}_3\text{-N}$ yang berkesan dari air sisa getah asli

oleh bahan penjerap komposit adalah 3g bahan penjerap, pH 7, kelajuan goncangan 150 rpm dan 100 min sentuhan masa. Penyingkiran maksimum bagi warna, COD dan $\text{NH}_3\text{-N}$ menggunakan bahan penjerap komposit dari air sisa getah asli masing-masing ialah 98%, 87.2% dan 94.2%. Siasatan model isoterma penjerapan menunjukkan bahawa data isoterma penjerapan sesuai dengan isoterma Langmuir. Ini menunjukkan bahawa liputan ekalapisan pada bahan penjerap komposit adalah dominan. Sebaliknya, kinetik penjerapan menunjukkan pematuhan yang baik untuk model pseudo-tertib kedua yang menunjukkan bahawa kadar tindak balas jerapan yang dikawal oleh mekanisma tertib kedua (penjerapan kimia). Penjerapan turus menunjukkan bahawa penyingkiran warna, COD dan $\text{NH}_3\text{-N}$ boleh mencapai sehingga 99.99% pada kadar aliran rendah 2 mL/min yang menghasilkan penyingkiran parameter yang lebih baik dan tempoh keletihan yang lebih perlahan. Sementara itu, data uji kaji didapati mematuhi model Thomas dan Yoon-Nelson berbanding dengan model Adam-Bohart. Analisa penjerapan/penyaherapan telah mencapai tiga (3) kitaran kebolegunaan bahan penjerap komposit. Kesimpulannya, kajian ini telah membuktikan bahawa bahan penjerap komposit adalah bahan penjerap yang berpotensi dalam penyingkiran warna, COD dan $\text{NH}_3\text{-N}$ dari air sisa getah asli.



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LIST OF SYMBOLS

C	-	Solute/outlet concentration
C_b	-	Effluent concentration
C_{ad}	-	Different between inlet/initial and outlet equilibrium concentration
C_{de}	-	The concentration of adsorbate desorbed
C_e	-	Concentration of adsorbate at equilibrium
C_i	-	Constant for Intra-particle diffusion model
C_t	-	The concentration of adsorbate at the time, t
C_0	-	Initial/inlet adsorbate concentration
K_F	-	Adsorption or distribution coefficient for Freundlich isotherm
K_L	-	The rate of adsorption for Langmuir isotherm
k_{pi}	-	Adsorption rate constant for intra-particle diffusion model
k_1	-	Adsorption rate constant for pseudo-first-order
k_2	-	Adsorption rate constant for pseudo-second-order
NH_3-N	-	Ammonia nitrogen
n	-	Constant for Freundlich isotherm
pH_{pzc}	-	Point of zero potential
Q_0	-	Adsorption capacity for Langmuir isotherm
q_e	-	Amount of adsorbate adsorbed per unit mass of adsorbent at equilibrium
q_t	-	Amount of adsorbate adsorbed per unit mass of adsorbent at the time, t
q_t, cal	-	Calculated adsorption uptake at the time, t
q_t, exp	-	Experimental adsorption uptake at the time, t
R_L	-	Separation factor
R^2	-	Coefficient of determination
S_{BET}	-	BET surface area
T	-	Time

V_{meso} - Mesopore volume

V_T - Total pore volume



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LIST OF ABBREVIATION

ANOVA	-	Analysis of variance
APHA	-	American Public Association
BET	-	Brunauer-Emmett-Teller
CH ₃ COOH	-	Acetic Acid
EBCT	-	Empty Bed Contact Time
EFB	-	Empty Fruit Bunch
FTIR	-	Fourier Transform Infrared
MTZ	-	Mass Transfer Zone
NH ₃ -N	-	Ammoniacal Nitrogen
RO	-	Reverse Osmosis
SEM	-	Scanning Electron Microscopy
XRD	-	X rays Diffraction
XRF	-	X ray Fluorescence

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CHAPTER 1

INTRODUCTION

1.1 Introduction

Malaysia is the fourth biggest rubber manufacturer in the world after Thailand, Indonesia and India. Rubber industry is one of the most important industries and plays a vital role in the Malaysia economy (Mokhtar *et al.*, 2015; Vijayaraghavan *et al.*, 2008). Natural rubber is an important raw material for production of more than 40,000 consumer products such as conveyor belts, rubber rollers and others, automotive products (fan belts, radiator hoses and others), latex products (rubber gloves, toys hygienic products) and many kinds of adhesives tires (major consumer), shoes, gloves and also medical devices (Nazri *et al.*, 2015; Pillai & Girish, 2014).

The raw material for natural rubber production is white fluid called 'Latex' which is obtained from *rubber Hevea Brasiliensis* trees (Ashok *et al.*, 2015). There are two types of processes in raw natural rubber processing which are; the production of concentrated natural rubber latex (NRL) and the standard Malaysian rubber (Mokhtar *et al.*, 2015). However, the production process of the rubber industry always leads to generate of a large amount of contaminated wastewater, as a result from a large quantity of water needed during processing (Mokhtar *et al.*, 2015; Pillai & Girish, 2014).

Rubber-processing industries generate wastewater in the range of 10 to 30 L/kg of raw material. This wastewater includes wash water, small amounts of uncoagulated latex, and serum with small quantities of protein, carbohydrates, lipids, carotenoids, and salts (Ashok *et al.*, 2015; Nazri *et al.*, 2015; Pillai & Girish, 2014). In general, rubber wastewater has a high concentration of $\text{NH}_3\text{-N}$, BOD, COD, nitrate, phosphorus and total solids (Rosman *et al.*, 2014; Kumlanghan *et al.*, 2008). The characteristics of natural rubber wastewater as follows: pH, 3.7 to 5.5; total suspended solids (TSS), 200

to 700 mg/L; biochemical oxygen demand (BOD₅), 1500 to 700 mg/L; and chemical oxygen demand (COD), 3500 to 14000 mg/L; total nitrogen (TN), 200 to 1800 mg/L and sulphate, 500 to 2000 mg/L (Mokhtar *et al.*, 2015).

High level of nitrogen and ammonia wastewater is discharged directly into surface waters wells, streams, lakes or even the sea, which will inevitably pollute the water. The discharge of these effluents into public water bodies can give rise to serious depletion of dissolved oxygen, thus affecting the normal environment supporting the aquatic system and lead to the death of some aquatic organisms living in the water (Pillai & Girish, 2014; Rosman *et al.*, 2014). Therefore, treatment of rubber wastewater using effective technologies for overcoming these problems is needed (Smitha *et al.*, 2012).

Environmental Quality (Industrial Effluents) Regulations 2009 under standard A and standard B is stringent environmental regulation for the control of rubber effluents which is enforced in Malaysia. Standard A for discharge upstream of any raw water intake, and standard B for discharge downstream of any raw water intake (Mamun & Zainudin, 2013). Each wastewater treatment plant must comply with these standards. Both standards consist of discharge temperature, pH, biological oxygen demand (BOD), chemical oxygen demand (COD), suspended solids (SS) and other heavy metals, but with different parameter limits (Mokhtar *et al.*, 2015).

Several technologies have been developed to treat natural rubber wastewater including such as anaerobic-cum-facultative lagoon system, anaerobic-cum-aerated lagoon system, aerated lagoon system and oxidation ditch system, aerobic granular sludge technology (Rosman *et al.*, 2014; Rosman *et al.*, 2013; Xin *et al.*, 2013; Abdullah & Sulaiman, 2013). However, these systems are inefficient for the removal of recalcitrant organics and micro-pollutants in natural rubber wastewater (Ashok *et al.*, 2015). In addition, these systems required large space, longer effluent treatment period, odour problems, high operating, and maintenance costs. These circumstances lead to frequent non-compliance with the legal discharge limits (Rosman *et al.*, 2014).

Therefore, the need to find effective and economical alternative techniques is taken as a priority in the water and wastewater treatment industry (Simate & Ndlovu, 2015; Barakat, 2011). Recently, adsorption has become one of the successful alternative treatments of wastewater (Barakat, 2011; Wan Ngah *et al.*, 2011). Adsorption is considered to be an efficient and versatile method, due to various adsorbents having good selectivity toward different types of wastewater,

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